



Examination of a Hot-Work Tool Steel Treated with Different Nitriding Processes after Cyclic Heating

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Abstract

This research examines the effect of salt bath, and nitriding processes on hot forming tool steel. The chosen steel is BÖHLER W350 ISOBLOCK hot forming tool steel, which is widely used in the industry for pressure casting. After surface treatment, some samples were exposed to cyclic heat and corrosion effects in a molten aluminium medium, thus simulating the conditions experienced during pressure casting. Microhardness measurements were performed on the samples treated with different procedures. The results were compared using a scanning electron microscope and energy-dispersive X-ray spectrometry.

Keywords: *plasma nitriding, salt bath nitriding, hot work tool steel.*

1. Introduction

Today, aluminium castings account for a significant proportion of automotive components. Aluminium is a light metal that enables the production of light machine parts without deterioration of their strength characteristics.

In the production of these parts, the casting tool plays a prominent role: even when used at high temperatures, it must show high resistance to both mechanical and corrosion effects. The industry uses hot-forming tool steel as the raw material for casting tools, which retains its heat resistance even at the melting point of aluminium. In order to preserve the quality of the tool in the long term, its surface treatment is essential.

Nitriding is a thermochemical surface treatment during which nitrogen is diffused into the surface of the workpiece in order to create a hard, wear-resistant layer containing nitrides [1, 2]. Many versions of nitriding are used in different industries in accordance with the requirements of the given area of use.

Previous research has reported an improvement in the thermal fatigue life of 1.2343 and 1.2344

steels after various nitriding treatments [3, 4]. Yucel Birol [5] investigated the effect of plasma nitriding against thermal fatigue for 1.2365 steel. During the thermal fatigue test between 450 °C and 750 °C lasting 500 cycles, the original surface hardness of the surface treated steel 1084 HV0.02 decreased to 250 HV0.02. The authors attributed the phenomenon to the thermal damage of the surface compound layer, the decrease in nitrogen content, and the microstructural transformation of the substrate.

In other research, Guang Chen et al [6] performed a similar experiment on steel number 1.2344 surface treated by salt bath nitriding at different temperatures (430, 450 and 470 °C) and treatment time (4, 6 and 8 hours). The specimens were held in molten aluminium at 750 °C for 30 minutes. As a result of their research, it can be concluded that the hardness values of the samples decreased from 1050-1317 HV0.1 to 855-891 HV0.1.

However, no literature data compares the two nitriding processes based on the results of thermal fatigue experiments performed under the same conditions. During our research, the cyclic

thermal fatigue test was performed on specimens treated with plasma nitriding and salt bath nitriding, and then the treatment procedures in terms of the decrease in hardness values and the change in nitrogen concentration were compared.

2. Research methodology

2.1. The examined steel and its treatments

The material examined during the research was BÖHLER W350 ISOBLOCK hot forming tool steel, whose chemical composition is shown in [Table 1](#).

The raw material was obtained as a steel bar with a cross section of 20×20 mm, then cut into 5 mm thick test pieces with a water-cooled abrasive cutting disc cutting machine. This type of steel is precipitation hardenable, so to reach its desired hardness, three annealing steps must be performed in its heat treatment cycle. The third annealing temperature coincides with the nitriding temperature, so the two processes were performed in one step. The preliminary heat treatment of the test specimens is shown in [Figure 1](#).

The salt bath treatment was carried out in a carbonitriding medium with a low cyanide content at 580 °C for 2.5 hours. Plasma nitriding was carried out at 520 °C in a 1:3 nitrogen and hydrogen gas mixture for 25 hours. During both technologies, the goal was to create a compound layer with a thickness of 10 µm and a diffusion layer with a thickness of 100 µm.

During aluminium pressure casting, molten aluminium at 650–700 °C was injected into the cavities of the casting tool, which often exceeded the speed of 30 m/s, at a pressure of up to 1300 bar [6]. The surface of the tool was exposed to strong

mechanical stress and severe corrosion effects. Damages caused by complex deterioration mechanisms are thermal fatigue, wear and corrosion.

During the research, the samples were subjected to cyclic heat and corrosion effects in a molten aluminium medium. The specimens were immersed in an aluminum medium at 700 °C for 5 minutes, then cooled in air for 5 minutes for 10 cycles.

After the surface treatment and corrosion testing, the samples were cut perpendicular to the surface. The mounted cross-section samples were sanded and polished. The samples have been given a name for ease of reference, as shown in [Table 2](#).

2.2. The test methods

Vickers hardness was measured on the samples in accordance with ISO 18203:2016, the standard for determining surface-treated crust thickness. First, the core hardness was measured from the average of three measurements taken in the middle of the samples.

After that, the additional measurement points were taken from the surface of the sample every 0.05 mm, in two measurement rows, offset by 0.025 mm from each other. A total of 20 measurements were made on one sample.

The mounted and polished samples were chemically etched with 2% Nital solution for 60–90 seconds, which made the microstructure visible and ready for further examinations.

Before the tests, performed with a Zeiss EVO MA 10 type scanning electron microscope (SEM), the surface of the sample embedded in the mounting resin was coated with a thin layer of gold, and then

Table 1. Chemical composition of the test specimens

C	Si	Mn	Cr	Mo	V
0.38	0.20	0.55	5.00	1.80	0.55

Table 2. Notations of the samples

Sample	Name
Reference	R1
Reference, cyclic heated	R2
Salt bath nitrided	S1
Salt-bath nitrided, cyclic heated	S2
Plasma nitrided	P1
Plasma nitrided , cyclic heated	P2

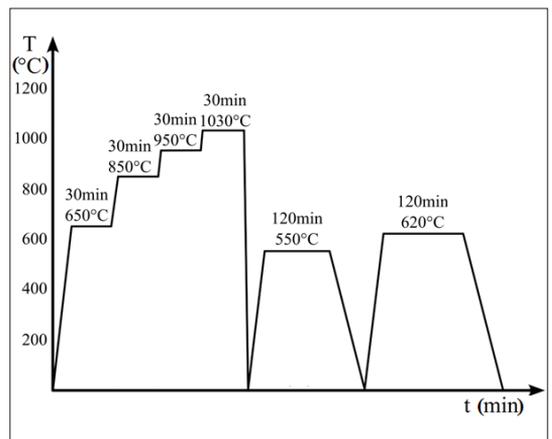


Fig. 1. Preliminary heat treatment of the samples.

the samples were attached to the sample holder with double-sided, carbon-based adhesive tape. The chemical composition of samples was determined by energy-dispersive X-ray spectrometry (EDS) during the electron microscopic examinations. During these tests, elemental analysis was performed on the individual samples, and the precipitates found on them, and the material quality of any compound layers that may have formed was determined.

3. The results of the tests

3.1. The results of the microhardness measurement

The results of the microhardness measurement are summarized in **Figures 2** and **3**.

Based on the results, it can be concluded that although the salt bath nitrided sample has a thicker diffusion zone, its hardness is lower than that

of the plasma nitrided sample. In the case of the samples treated in molten aluminium, a significant decrease can be seen in the hardness values. It was an average of 10% for the reference sample, an average of 6.3% for the salt-bath-treated sample up to a layer depth of 0.1 mm, and 17.8% for the plasma-nitrided sample..

3.2. The results of the examinations performed with a scanning electron microscope

Figure 4 shows the network structure formed in the case of the nitrided samples, which appears more strongly in the samples treated in the salt bath.

The white band visible on the surface of the samples was presumably the compound layer, but this can only be confirmed after elemental analysis. The nets appeared even more densely at the corners of the samples (**Fig. 5**).

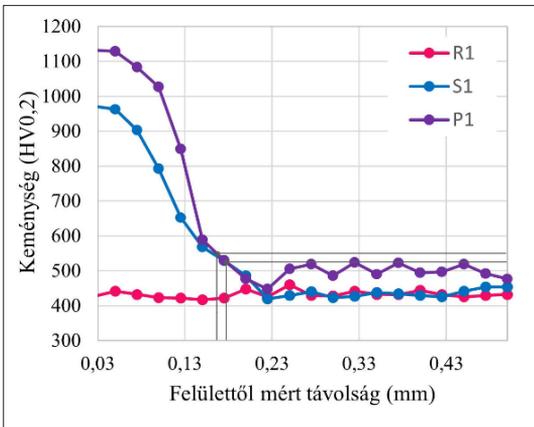


Fig. 2. Comparison of the results of each procedure before aluminium treatment.

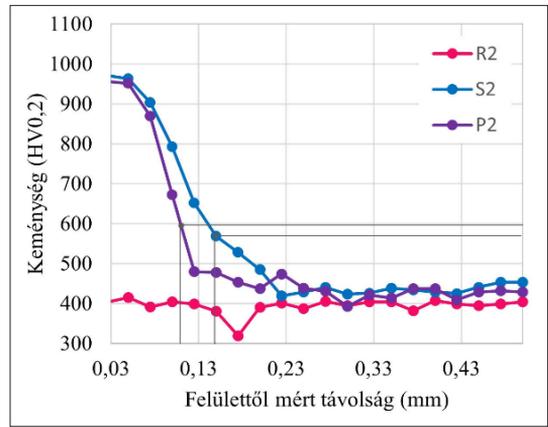


Fig. 3. Comparison of the results of individual procedures after aluminium treatment.

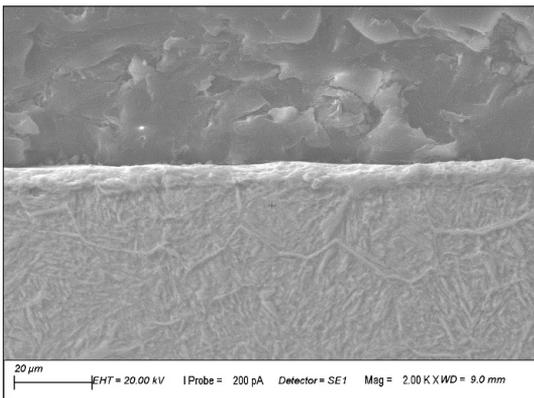


Fig. 4. Cross-sectional view of sample S1.

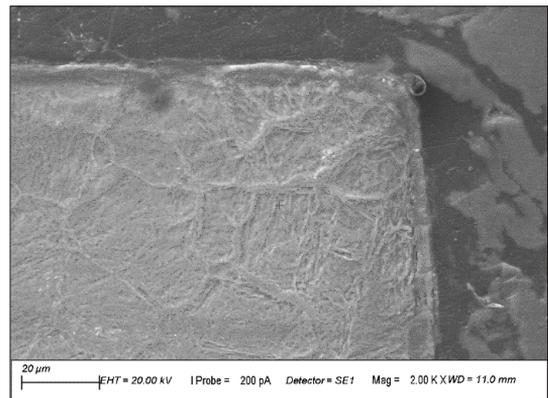


Fig. 5. Nitride structure formed on S2 sample's edge.

3.3. Results of energy dispersive X-ray spectrometry

Due to their high nitrogen content, the nitride structure visible on the sample's edges was nitride precipitation, which formed along the grain boundaries.

Along the corners, nitrogen was present in a higher mass percentage. This phenomenon can be attributed to the fact that at the corners of the samples, nitrogen has the opportunity to diffuse into the base material from several directions, due to the resulting local supersaturation, the excess amount of nitrogen appears in the form of precipitates along the grain boundaries [7].

This so-called edge effect was primarily observed during plasma nitriding. However, a higher nitrogen content can be detected in each nitrided sample. Based on the results of the EDS test, in the case of the plasma nitrided samples, this amount was almost twice the nitrogen content of the samples treated in the salt bath.

The corners of the nitrided samples are presumably harder due to the edge effect, but they are more brittle. This phenomenon was not desirable in all cases, as the hardened material can easily break off with tools subject to dynamic stress. However, due to their good tribological properties, hard edges have a good resistance to wear. During examination of the top surfaces of the nitrided samples, it was found that their nitrogen content was almost the same. A possible reason for this was that although the two processes were performed with different production parameters, the goal was to achieve the same diffusion and compound layer thickness in both cases. Nitrided samples with index 2 show a significant decrease in nitrogen content. The values measured on different samples and areas are illustrated in **Table 3**.

Based on the results, it can be concluded that while the nitrogen content of the salt bath samples decreased by only nearly 40%, the plasma

nitrided samples lost a much more significant amount of nitrogen, their nitrogen content decreased by 66–67%.

This phenomenon can be explained by the fact that the ϵ -nitrides formed during salt bath nitriding are presumably thermodynamically more stable and have better heat resistance [8].

The statement is also supported by the results obtained during the hardness measurement, where, in the case of the sample treated in the salt bath, the hardness level decreased to a much lesser extent because of heat cycling.

During decomposition, diffusion processes start towards areas poorer in nitrogen, which was the environment surrounding the test pieces. As a result, the nitrided layer became depleted in nitrogen. In contrast to the reference sample, complete dissolution of the base material or the nitrided layer on the surface of the test specimen that underwent both plasma and salt bath nitriding was not discovered.

4. Summary of results

The microhardness measurement tests determined the different hardnesses and layer depths achieved by each nitriding process. It can be concluded that the surface hardness of the plasma nitrided sample is higher, but the thickness of the formed layer is somewhat smaller than in the case of samples produced by the salt bath process.

After cyclic treatment in the aluminium bath, the electron microscopic examination showed that the nitrided layer protected both nitrided samples from the formation of intermetallic compounds. At the same time, in the case of both treatments, a significant change can be observed in the hardness of the samples and the thickness of the nitrided layer. In this regard, it can be concluded that after treatment, the sample nitrided in the salt bath suffered less damage in terms of both hardness and layer thickness during the cyclic heat load.

The elemental analysis supported the assumptions made in previous measurements, and we were able to make further findings regarding the composition of the samples. From the high nitrogen concentration of the nitride structure located on the edge of the samples, it can be concluded that the edge effect mainly affects plasma-nitrided samples.

In the case of nitrided samples suffering from corrosion in molten aluminium, we could make the statement that the sample treated in a salt

Table 3. Change in nitrogen concentration of individual samples

Sample	Nitrogen content (m/m%)		
	1	2	Δ (%)
S (on top surface)	4.6	2.8	39.1
S (corner)	5.7	3.5	38.6
P (on a top surface)	5.8	1.9	67.2
P (corner)	10.5	3.6	65.7

bath loses its nitrogen content to a lesser extent than the plasma nitrided piece. This was explained by the different thermodynamic stability of the different nitrides and the diffusion processes following the decomposition.

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